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A Three Dimensional Analysis of Au-Silica Core-Shell Nanoparticles Using Medium Energy Ion Scattering

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Motivation and background

Metallic nanoparticles (NPs) with or without a dielectric shell are potential candidates for many applications, e.g. in plasmonics, catalysis, or healthcare. Since the physical, chemical and geometrical properties are usually strongly linked, the tailoring of these properties on the nanoscale is of fundamental importance. The aim of this work is two fold:

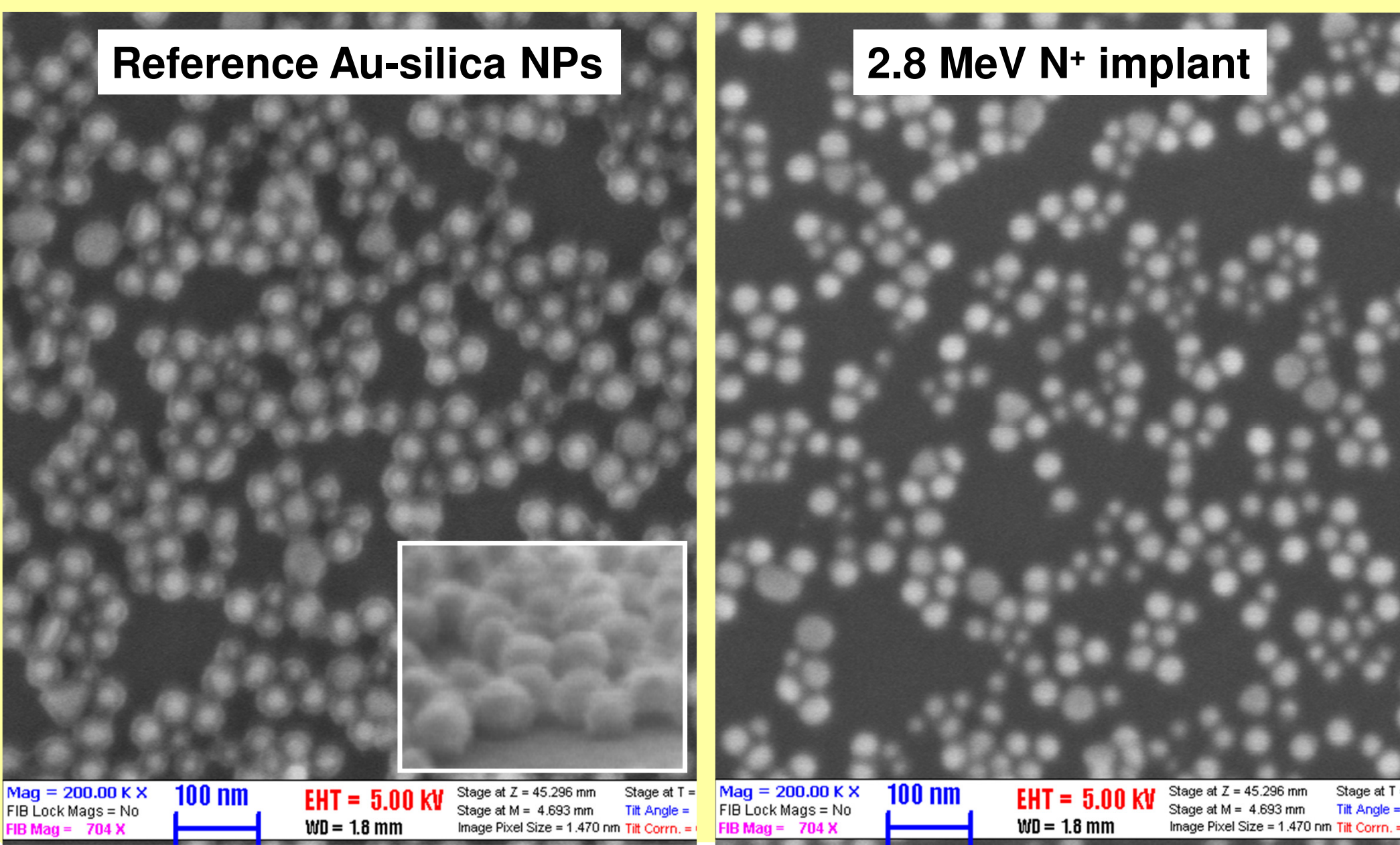
- To demonstrate the capability of the **Medium Energy Ion Scattering** (MEIS) technique, combined with a **3D spectrum simulation** tool (RBS-MAST code [1]) to determine the shape, size, and atomic composition of Au-core silica-shell particles on the nanoscale.
- To better understand ion beam-induced structural and compositional changes of dielectric and metallic nanoparticles. The high-energy **ion irradiation-induced shaping** of dielectric and metallic nanoparticles has been previously reported [2, 3]. However, low energy ion implantation may be a potential nanofabrication tool for the doping and alloying of the particles. In this work we exposed spherical Au-core silica-shell nanoparticles to ion irradiation with a wide range of different parameters (ion mass, ion energy): 30 keV Ar⁺, 150 keV Fe⁺, and 2.8 MeV N⁺ irradiation. The geometrical changes were monitored using MEIS measurements combined with 3D spectrum simulation.

Experimental details

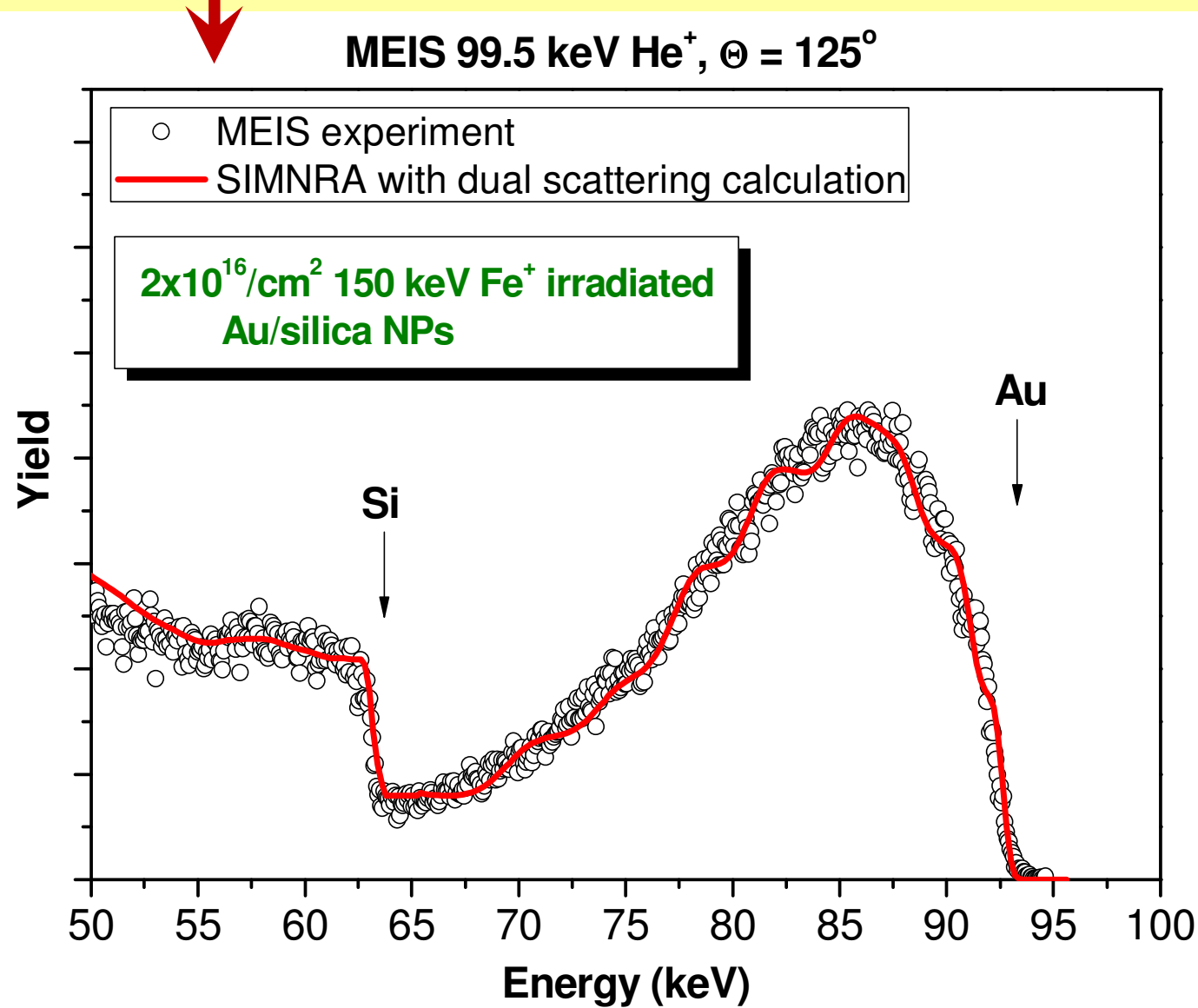
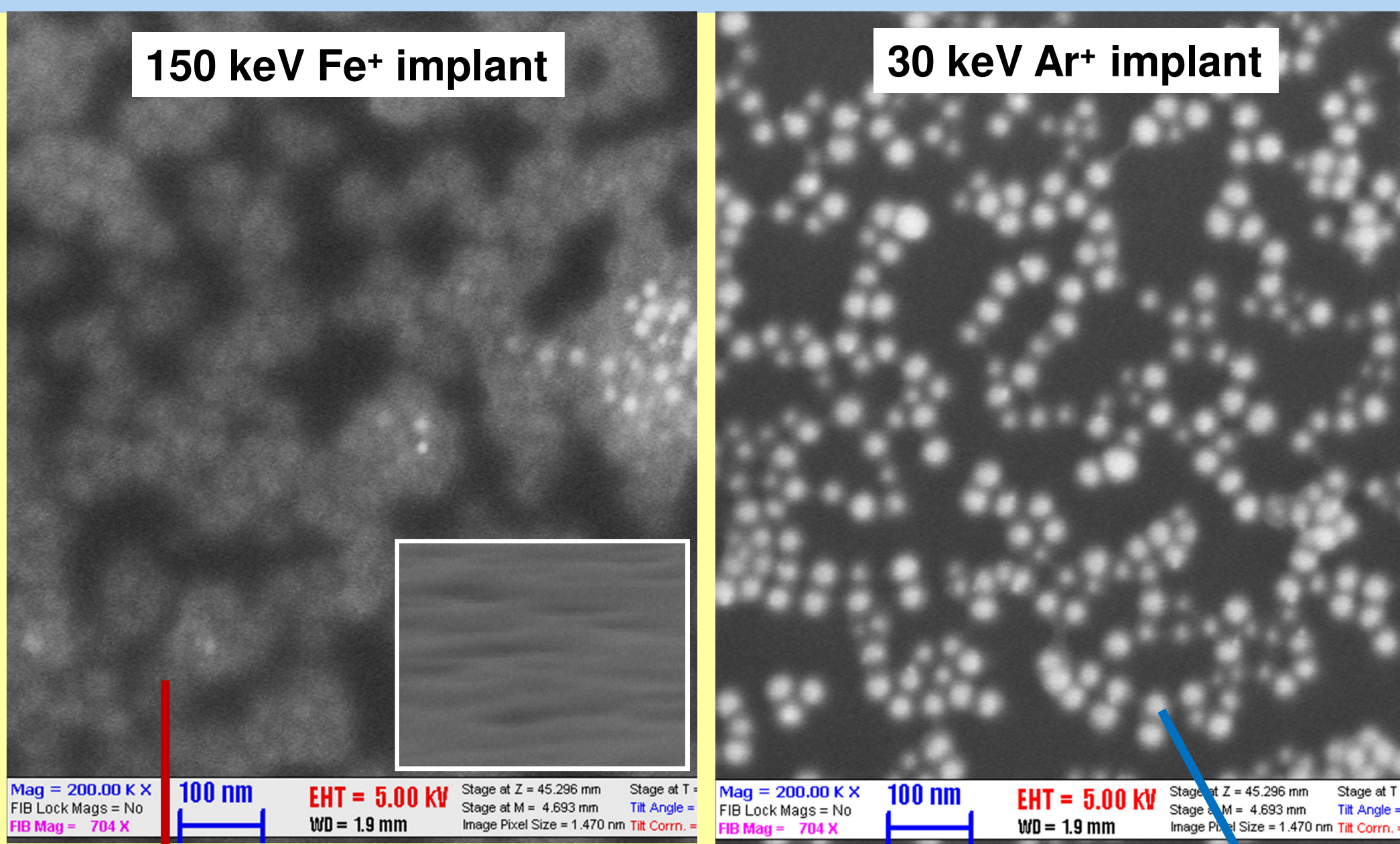
- Samples:** Au-silica core-shell nanospheres (core $\varnothing \sim 25$ nm, shell $\varnothing \sim 40$ nm) were deposited on Si(100) substrates using the Langmuir-Blodgett (LB) technique. A thin, colloidal Stöber silica shell was grown on the Au cores with a sol-gel technique. Planar gold layers deposited on glass substrates were also characterized for comparison.
- MEIS** analysis was performed at the IIAA Huddersfield using 100 keV He⁺ ions scattered through angles of $\Theta = 90^\circ$ and $\Theta = 125^\circ$. Backscattered ions were detected with a toroidal electrostatic analyser.
- FESEM** analysis was carried out with a LEO 1540 XB microscope at MTA EK MFA in Budapest.

MEIS spectrum simulation

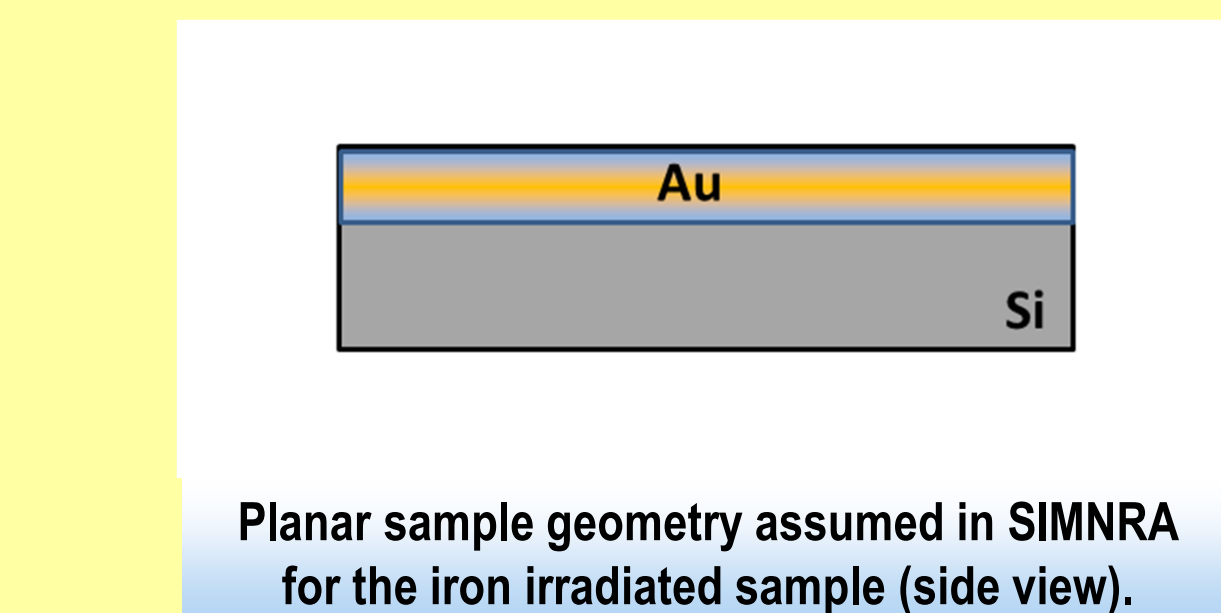
The **RBS-MAST** code is used to reconstruct the spectrum shape according to the 3D structure of the sample (without straggling contributions). The **DEPTH** [4] code is applied to calculate the energy resolution due to straggling, multiple scattering and system resolution. The **SIMNRA** [5] code is used to calculate the dual scattering contribution to the overall MEIS energy spectrum.



FESEM images of Au-silica nanospheres. The insets show side views of the unirradiated and iron irradiated sample. Iron ions cause significant morphology change leading to a quite smooth surface due to strong recoil effects which moves the Au atoms into the Si substrate.

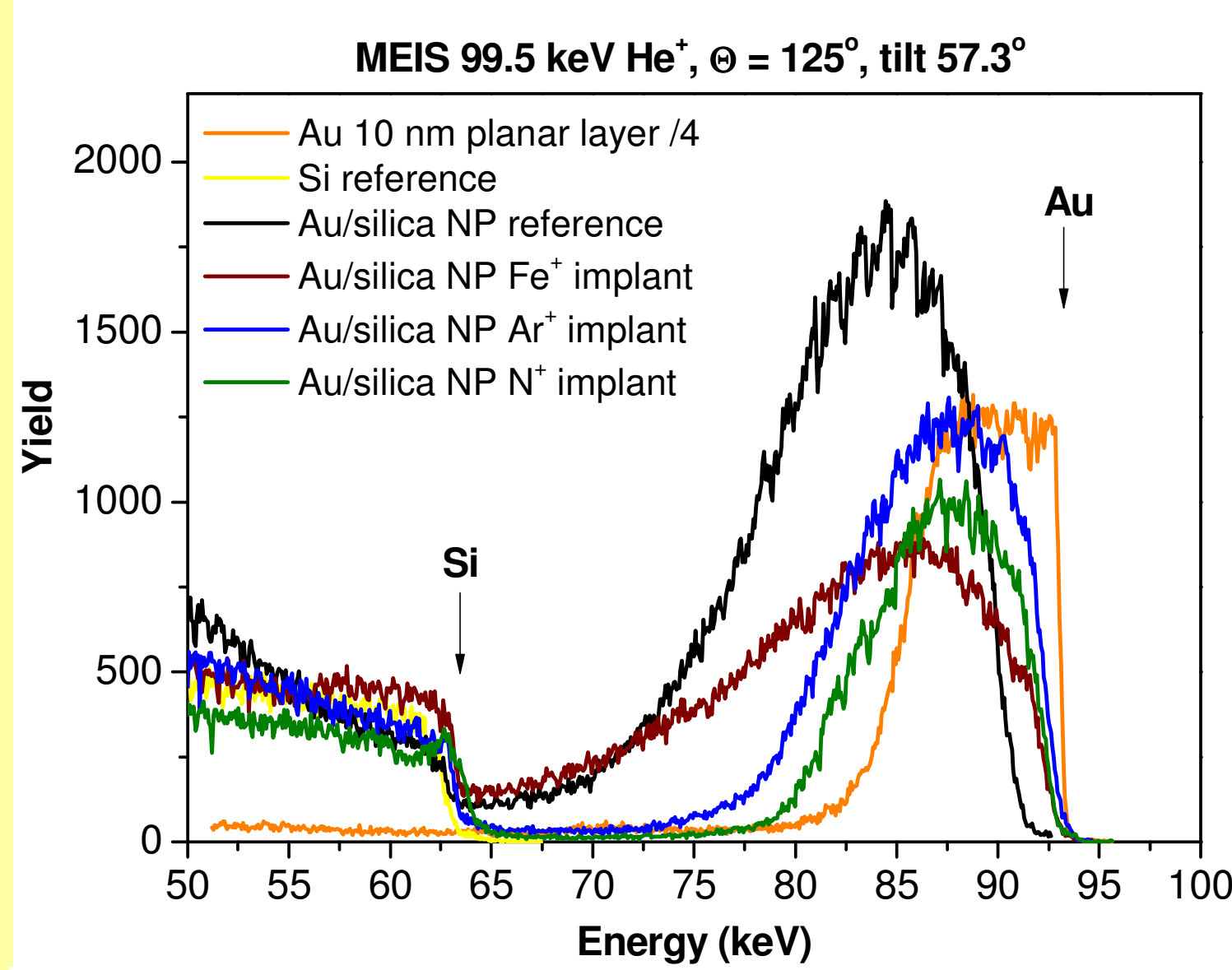


The broadening of the Au signal for Fe⁺ bombardment indicates different relocation processes on the nanoscale than observed for Ar⁺ and N⁺ irradiation.

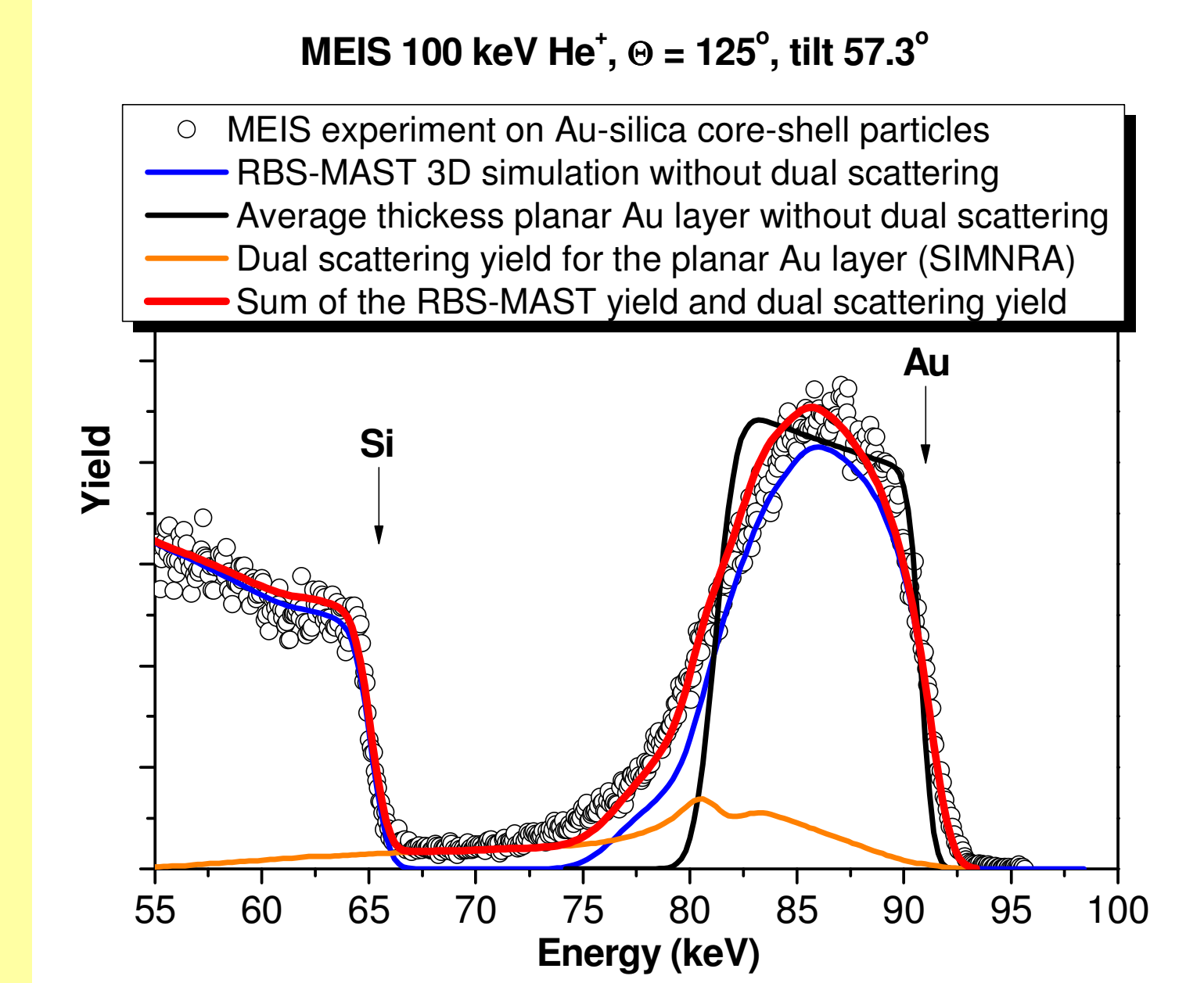
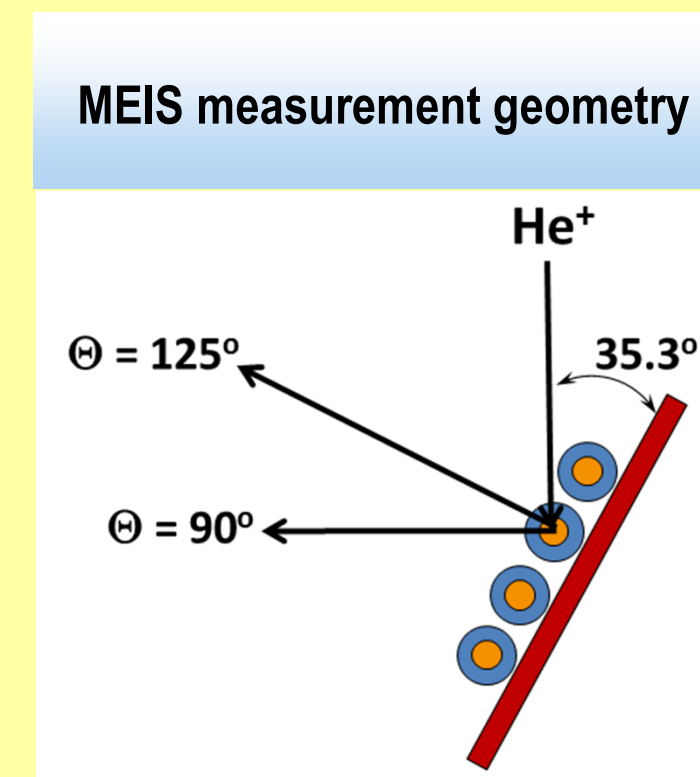
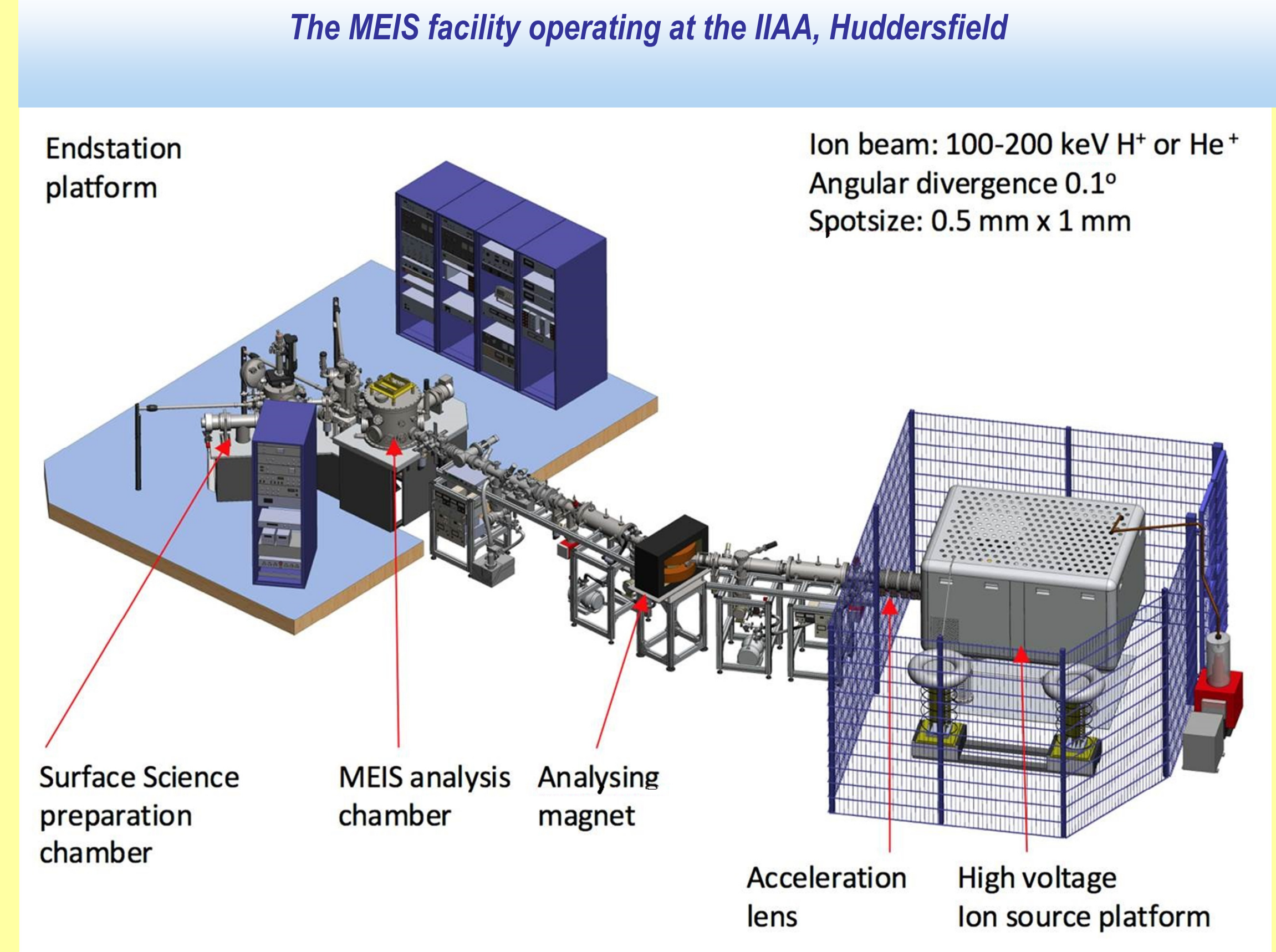
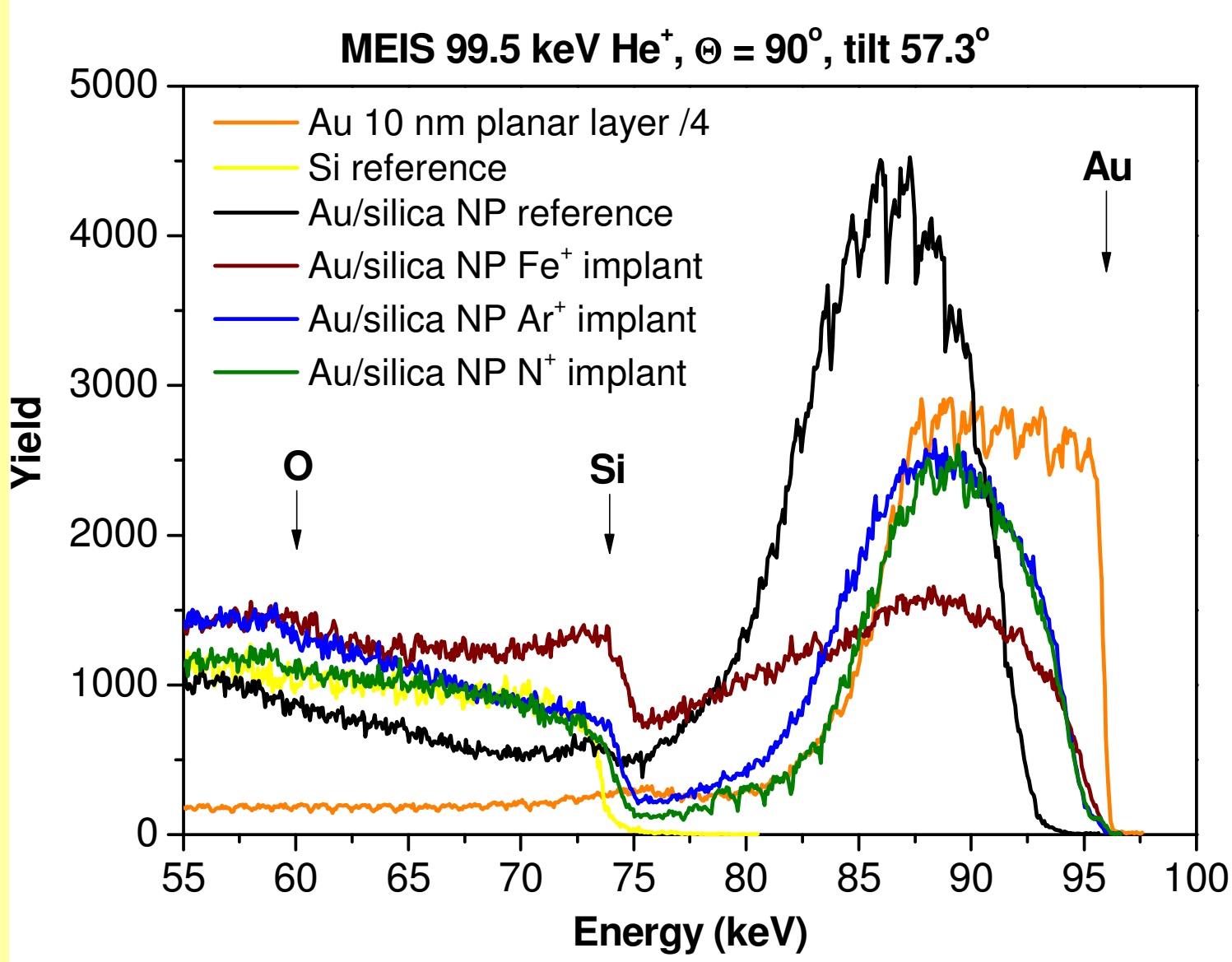


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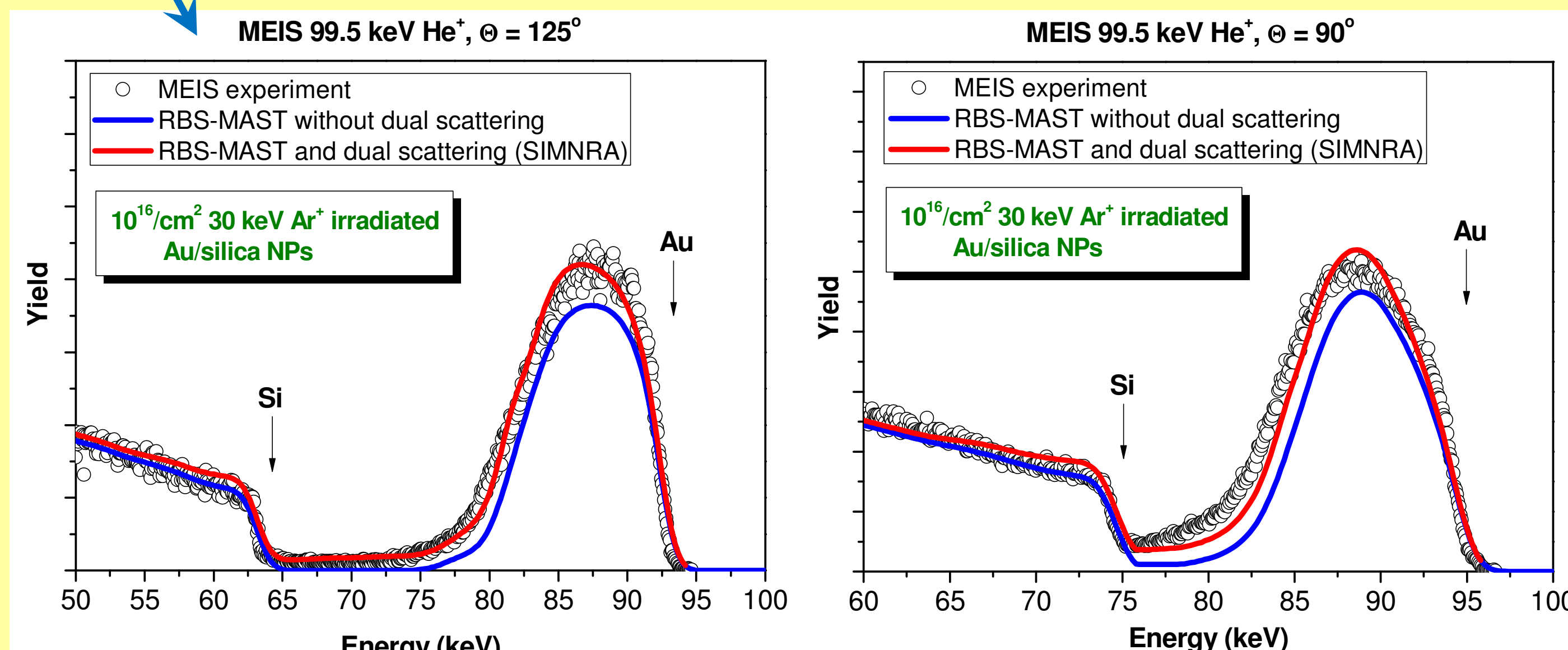
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- [2] Z. Zolnai, Appl. Surf. Sci. **281** (2013) 17; Z. Zolnai *et al.*, Phys. Rev. **B 83** (2011) 233302
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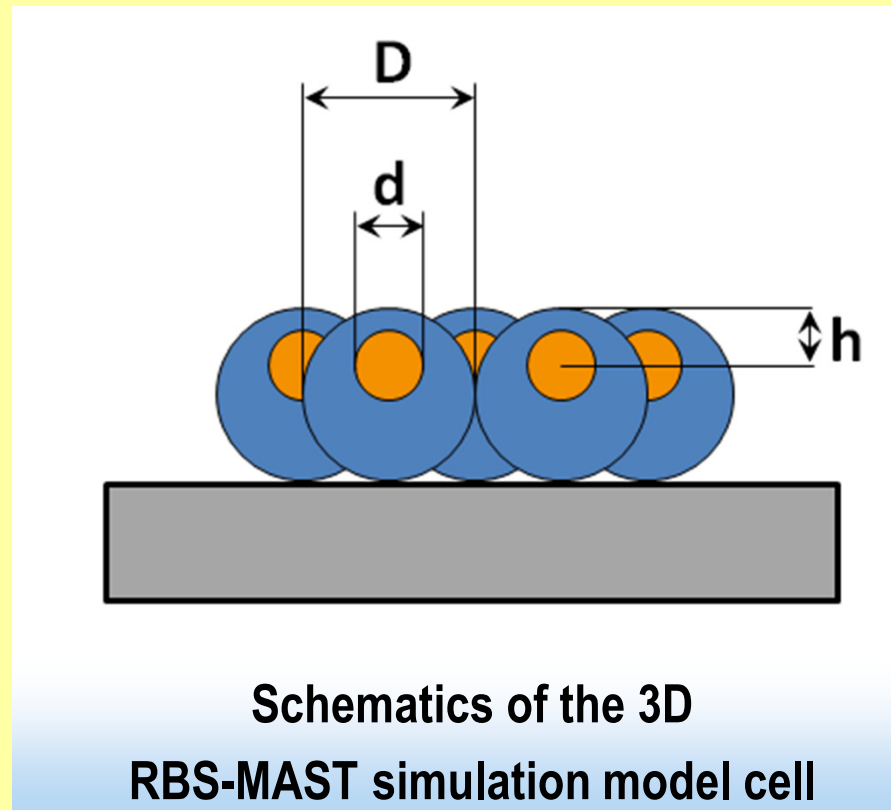
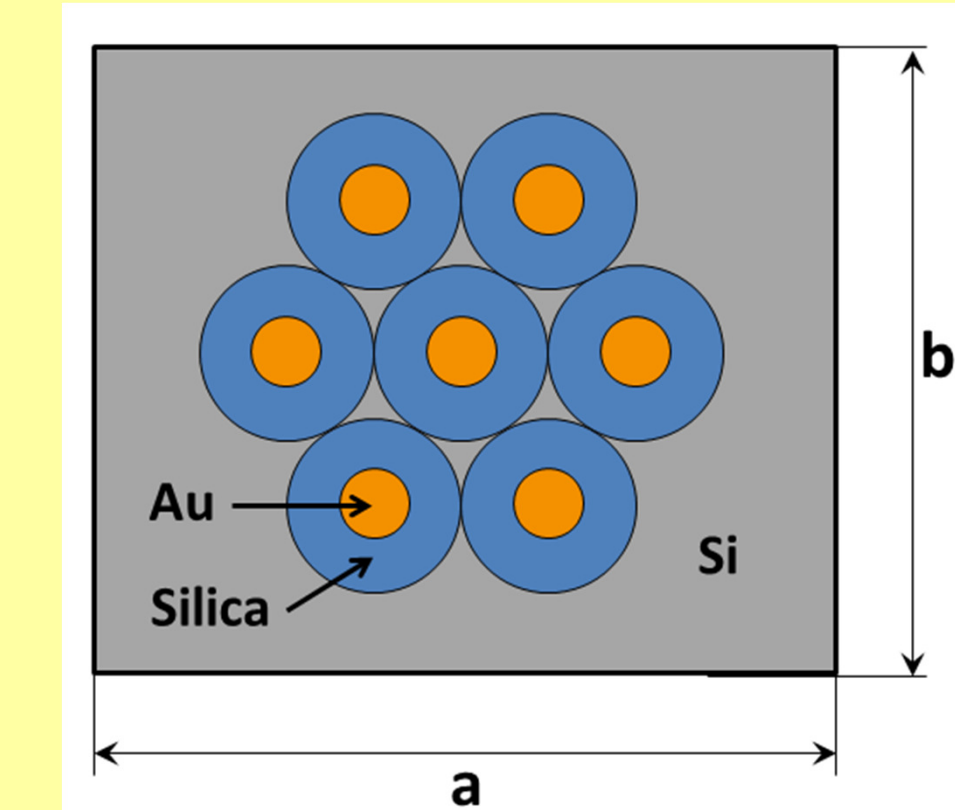
MEIS spectra of unirradiated and irradiated Au/silica NPs together with that of a planar Au layer for comparison.



An example of the dual scattering contribution appearing as a background in the MEIS spectra of unirradiated (reference) Au/silica NPs.

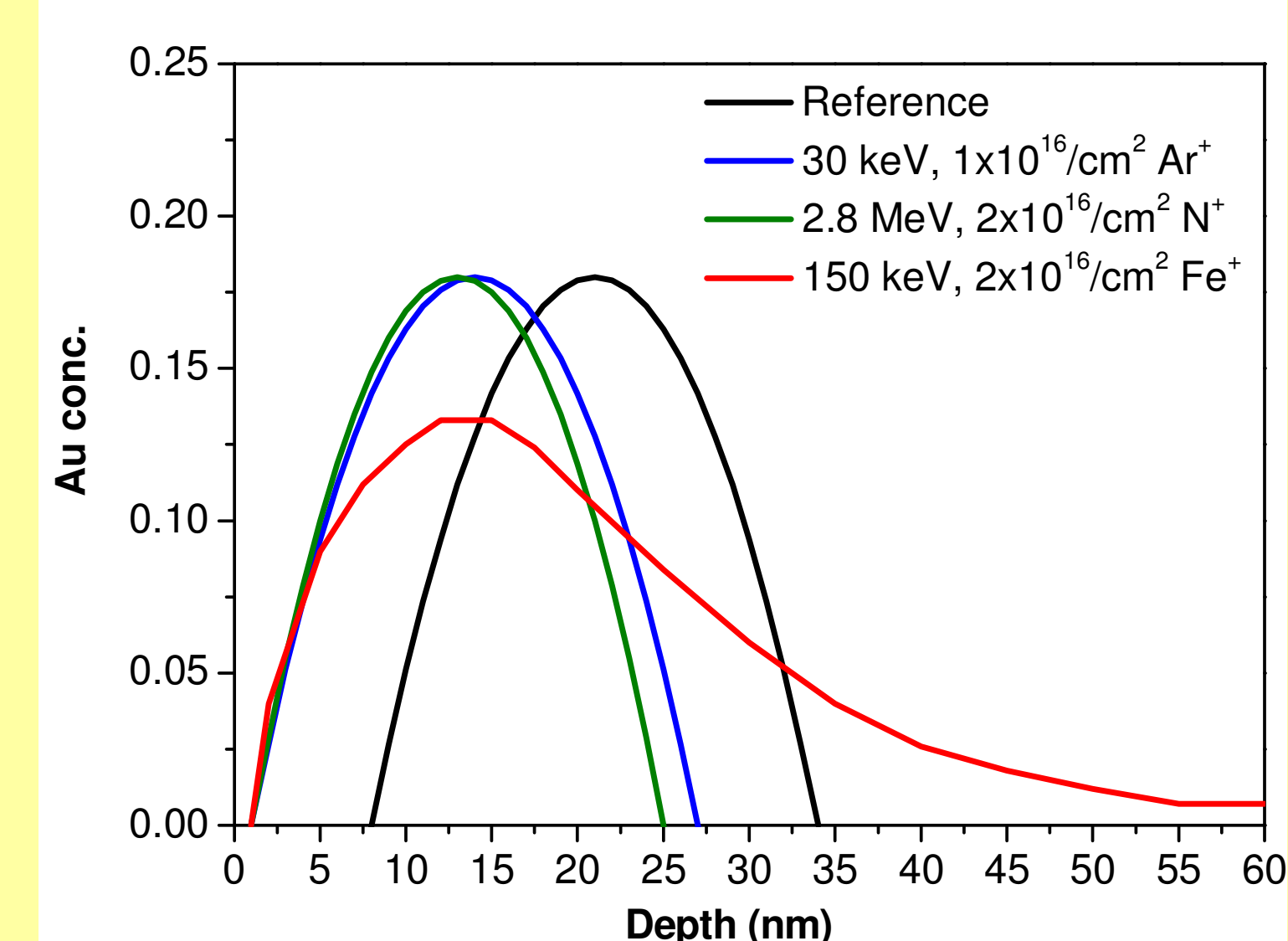


Argon irradiation causes the removal of the front part of the silica shell through sputtering of Si and O atoms. The spherical Au core is still present but it gets closer to the surface of the nanoparticle to within 1 nm.



Cell parameters

- a = 214 nm
- b = 185 nm
- d = 26 nm
- D = 34 nm
- h = 14 nm



Schematic Au depth profiles as evaluated from MEIS spectra for various ion irradiation parameters. As an interesting result, despite the big difference in the irradiation conditions for low energy Ar⁺ and high energy N⁺ ions, their effect on the NPs is quite similar.

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